



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: M.D. Vrbanac et al.

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Examiner: P. Chin

Title: GRANULAR POLYSACCHARIDE HAVING  
ENHANCED SURFACE CHARGE

INVENTOR'S DECLARATION PURSUANT TO 37 C.F.R. § 1.132

TO THE COMMISSIONER FOR PATENTS:

I, Sherry L. Thomson, declare as follows:

1. I am a co-inventor named in the above-identified patent application. My educational experience and work background are as follows: I received a Bachelor of Science Degree in Chemistry from Washington State University in 1991 and a Master of Science in Pulp and Paper Science from the University of Washington in 1996. Since 1992, I have been employed by Weyerhaeuser Company as a Project Specialist.
2. I have read the Examiner's Office Actions dated January 4, 2001 and July 17, 2001 in Application No. 09/421,621, which is the parent application of the above-identified application.
3. To evaluate and compare the surface charge of modified starch particles representative of the teachings of the present invention and the surface charge of flocculated starch particles representative of the teachings in Example 3 of the Pruszynski reference (U.S. Patent No. 5,942,087) cited by the Examiner, I conducted the following experiments.
4. Materials. Five grams of dry starch (Douglas X-18 that was partially modified for starch cookout and was at 89.1% solids) was weighed in a beaker. After weighing the starch, deionized water was added to the beaker to form a starch slurry. The beaker was only partially filled to allow for the addition of the pre-treatment polymers solutions. To prepare the polymer solutions, each of the four polymers identified below were diluted to 0.1% solids by weighing

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LAW OFFICES OF  
CHRISTENSEN O'CONNOR JOHNSON KINDNESS<sup>PLLC</sup>  
1420 Fifth Avenue  
Suite 2800  
Seattle, Washington 98101  
206.682.8100

0.5 grams in a beaker and then diluting the polymer to 500 mL with deionized water. Each polymer solution was mixed with a hand blender for approximately fifteen seconds. For these experiments, the polymers that were: Nalco 7527 (cationic, coagulant, high charge, low molecular weight), Nalco 5200 (cationic, coagulant, high charge, low molecular weight), Nalco 7520 (cationic, flocculant, low charge, high molecular weight), and Nalco 7535 (cationic, flocculant, low charge, high molecular weight). When the experiments where performed, a pre-determined amount of a polymer was added to a beaker that contained a starch slurry. For each experiment, only one of the polymers was added. In addition, the specific dosages added were 0.66 lb/ton and 5 lb/ton as received. However, one of the flocculants, Nalco 7535, was also tested at a dosage of 1.33 lb/ton as received.

5. To perform the experiments, a polymer solution was added while a starch solution was still on the balance; the amounts added were, 12.5 mL (5 lb/ton), 3.3 mL (equivalent to 1.33 lb/ton), or 1.65 mL (equivalent to 0.66 lb/ton). After addition of the polymer, deionized water was added to obtain a total volume of 50 mL (approximately 50 g). After diluting with the deionized water, each starch slurry was mixed with a stirring rod; however, mixing was minimized to not impose excessive shear. In conjunction with these polymer addition experiments, a blank of slurred starch was also prepared without any polymer. This blank was also diluted with deionized water. In contrast to the invention of the present application, the Pruszynski U.S. Patent No. 5,942,087 describes (at column 5, lines 45-48) the use of a flocculant that is a cationic latex copolymer of acrylamide and dimethylaminoethylacrylate quaternized with methyl chloride (80/20 mole %). The commercially-available flocculant most similar to the polymer described in the Pruszynski reference is the cationic flocculant Nalco 7535. In addition, Nalco 7520 is another commercially-available cationic flocculant polymer that is also similar to the polymer described in the Pruszynski reference.

6. Test Methods. After addition of the polymers to the starch slurry samples, each sample was placed on a separate microscope slide with a cover glass that was carefully placed on top to avoid disturbing any starch flocculation. After placement of the cover glass, each sample was viewed under a microscope having a digital camera attached. For each sample, a picture of the starch was taken using the digital camera. A camera lens was chosen to accommodate the different sizes of the starch particles. After the pictures were taken, a computer program in

Microsoft Excel was used to measure the area of the starch particles and floc size. For each sample the area of at least 100 particles was counted.

7. In addition to measuring starch particle size, the particle surface charge was measured. To measure particle charge, the starch samples with polymer were diluted 2:1 with deionized water and then run on a Mutek PCD02 streaming current detector. During the runs, the initial voltage reading was recorded. If the voltage reading was unstable, a second sample was tested.

8. Test Results. The particle area results are provided in Table 1. These results demonstrate that the particle sizes of the starch with flocculant were higher than the particle sizes of the starch with coagulant. Although there is overlap in the particle size data with coagulant Nalco 5200, this overlap is an artifact of additional shear that was necessary to add to the flocculant treated starch to break apart the starch particles sufficiently in order to obtain a measurement. Qualitatively, coagulants formed very different starch slurries than the flocculants. At the highest dosage, the flocculant treated starch formed a large mass of starch particles that was difficult to break apart while mixing. In contrast, the coagulant treated starched formed small discrete particles. In addition, the images of the starch particles were more indicative of the smaller discrete particles of the coagulant treated starch whereas the flocculant treated starch particles were large agglomerates.

Table 1. Starch particle size.

Polymer Type	Chemical Designation	Dosage lb/ton As Received	Particle Area $\mu\text{m}^2$	Std. Dev.	Minimum $\mu\text{m}^2$	Maximum $\mu\text{m}^2$	NOTES
Coagulant	Starch Only	0	62	73	6	755	
	Starch Only	0	70	77	3	637	
Flocculant	Nalco 5200	0.66	489	784	5	4266	
	Nalco 7527	0.66	463	587	10	3970	
Coagulant	Nalco 7520	0.66	374	569	3	3150	
	Nalco 7535	0.66	651	2370	9	26190	
	Nalco 7535	1.33	2525	6449	1	39759	
Flocculant	Nalco 5200	5	1951	12387	10	208541	Duplicate
	Nalco 7527	5	702	1227	5	7050	
	Nalco 7527	5	570	864	13	6301	
Flocculant	Nalco 7520	5	2687	31731	0	710403	Highly flocculated-needed agitation
	Nalco 7535	5	1836	10598	3	195844	Highly flocculated-needed agitation

9. The starch particle charge readings are summarized in Table 2 below. As can be seen in Table 2, only the coagulant-treated starch particles achieved a stable positive charge at a typical coagulant dosage of 5 lb/ton. The coagulant polymers that were used, Nalco 5200 and Nalco 7527, are representative of the teachings of the present invention. At a dosage rate of 0.66 lb/ton, the starch treated with either of the flocculants had negative streaming current readings. At a dosage rate of 5 lb/ton, the starch treated with either of the flocculants had unstable streaming current readings.

Table 2. Starch particle charge.

Polymer Type	Chemical Designation	Dosage lb/ton As Received	Mutek1 mV	Mutek2 mV	NOTES
Coagulant	Starch Only	0	-370		Mutek reading drifted
	Starch Only	0	-400		Duplicate-Mutek reading drifted
Flocculant	Nalco 5200	0.66	-332		
	Nalco 7527	0.66	-299		
Coagulant	Nalco 7520	0.66	-312		
	Nalco 7535	0.66	-118		
	Nalco 7535	1.33	-379		
Flocculant	Nalco 5200	5	345		
	Nalco 7527	5	337		
	Nalco 7527	5	450		Duplicate
	Nalco 7520	5	-40	100	Highly flocculated-needed agitation, Mutek reading unstable
	Nalco 7535	5	-6	58	Highly flocculated-needed agitation, Mutek reading unstable

10. Conclusions. As demonstrated by the test results above in Table 2, the modified starch particles that are representative of the present invention exhibit stable positive charges whereas the flocculated starches representative of the teachings of Example 3 in Pruszynski exhibit unstable charges. In addition, as demonstrated by the test results above in Table 1, the charged modified starch particles representative of the present invention are considerably smaller in size than the flocculated starch particles representative of the teachings of Example 3 in Pruszynski. Furthermore, the modified starch particles that are representative of the present invention exhibited a stable positive charge and smaller particle size: the charge and small size allow addition of the modified starch particles into a normal papermaking system that uses normal retention aid strategies. Finally, qualitative observation of the differences between flocculant treated starch and the treatment in the present invention further substantiates that flocculant treatment forms large uncharged agglomerates whereas the present invention forms

small charge particles. When considering the differences of particle size and charge, it is apparent that flocculant treated starch relies only on a mechanical filtration mechanism of retention in papermaking. In contrast, the retention mechanism in papermaking with the present invention relies upon a charge mechanism of retention.

11. All statements made herein and of my own knowledge are true; and all statements made on information and belief are believed to be true. Further, these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the above-identified application or any patent issued thereon.

Date: 5-20-02

Sherry L Thomson  
Sherry L Thomson

LAW OFFICES OF  
CHRISTENSEN O'CONNOR JOHNSON KINDNESS<sup>PC</sup>  
1420 Fifth Avenue  
Suite 2800  
Seattle, Washington 98101  
206.682.8100